

TITLE OF INVENTION**PROCESS FOR PREPARING POLY(TRIMETHYLENE TEREPHTHALATE) FIBER**

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FIELD OF THE INVENTION

The present invention relates to a polyester yarn and its manufacture. More particularly, the invention relates to a process for producing poly(trimethylene terephthalate) fibers having good physical properties.

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BACKGROUND OF THE INVENTION

Polyethylene terephthalate ("2GT") and polybutylene terephthalate ("4GT"), generally referred to as "polyalkylene terephthalates", are common commercial polyesters. Polyalkylene terephthalates have excellent physical and chemical properties, in particular, chemical, heat and light stability, high melting points and high strength. As a result they have been widely used for resins, films and fibers.

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Polyesters prepared by condensation polymerization of the reaction product of a diol with a dicarboxylic acid can be spun into yarn. U.S. Pat. No. 3,998,042 describes a process for preparing poly(ethylene terephthalate) yarn in which the extruded fiber is drawn at high temperature (160° C.) with a steam jet assist, or at a lower temperature (95° C.) with a hot water assist. Poly(ethylene terephthalate) can be spun into bulk continuous filament (BCF) yarn in a two-stage drawing process in which the first stage draw is at a significantly higher draw ratio than the second stage draw. U.S. Pat. No. 4,877,572 describes a process for preparing poly(butylene terephthalate) BCF yarn in which the extruded fiber is drawn in one stage, the feed roller being heated to a temperature 30° C. above or below the Tg of the polymer and the draw roller being at least 100° C. higher than the feed roller.

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U.S. Pat. No. 6,254,961 relates to spinning poly(trimethylene terephthalate) into yarn suitable for carpets. According to this patent, drawing speeds of greater than 1000 m/min. are possible with the inventive process, with drawing speeds greater than 1800 m/min. desirable because of the high tenacity of the resulting yarn.

U.S. Pat. No. 6,284,370 relates to a poly(trimethylene terephthalate) fiber which has a suitable thermal stress and a suitable boil-off shrinkage and which gives a fabric, when woven or knitted, showing less stiffness caused by excessive shrinkage, and manifesting softness and the excellent color developing property expected from the low elastic modulus characteristic of the fiber. According to this reference, the intrinsic viscosity of a polymer used in the invention is preferably from 0.4 to 1.5, more preferably from 0.7 to 1.2. The polyester fiber of the invention preferably is in the form of multifilament yarn when used for clothing applications. Although the total size of the yarn is not restricted, it is usually from 5 to 200 d (denier), preferably from 20 to 150 d. Although the single filament size is not restricted, it is from 0.1 to 10 d, preferably from 0.5 to 5 d, more preferably from 1 to 3 d. Also according to this patent, it is important that the peripheral speed of a first roll used to produce the fiber be from 300 to 3,500 m/min. The peripheral speed is preferably from 800 to 3,000 m/min, more preferably from 1,200 to 2,500 m/min. Although the peripheral speed of a second roll is determined by the draw ratio, it is usually from 600 to 6,000 m/min.

U.S. Pat. Pub. No. 2003/0127766 relates, in general, to a poly(trimethylene terephthalate) BCF carpet modified cross-section yarn and a method for preparing the same and in particular, to a poly(trimethylene terephthalate) BCF carpet modified cross-section yarn and a method for preparing the same. According to this reference, poly(trimethylene terephthalate) with an intrinsic viscosity of 0.8 to 1.2 and a moisture content of 50 ppm or less is used as raw materials, and preferably melt-spun at a spinning rate of 1500 to 4000 m/min. Spun filaments are drawn at a rate of 1500 to 4000 m/min. and crimped.

U.S. Pat. Pub. No. 2003/0045611 relates to a process for preparation of pigmented shaped articles (e.g., fibers). For fiber use, poly(trimethylene terephthalate) preferably has an intrinsic viscosity that is about 0.6 dl/g or higher, and typically is about 1.5 dl/g or less. Preferred viscosities for many end uses, and, particularly for fibers and films, are 0.8 dl/g or higher, more preferably 0.9 dl/g or higher. Typically, the viscosity of poly(trimethylene terephthalate) fibers and films is 1.4 dl/g or less, 1.2 dl/g or less, or 1.1 dl/g or less. In commercial applications, the spinning speed is preferably at least about 1,000 meters/minute, and may be up to about 5,000 meters/minute or more, using roll 40 as reference speed.

SUMMARY OF THE INVENTION

According to a first aspect in accordance with the present invention a process comprises:

- (a) spinning molten poly(trimethylene terephthalate) polymer having a number average molecular weight of at least about 26500 and a melt viscosity of at least about 350 Pascals at 250°C and 48.65 per second shear rate;
- (b) converging the filaments into yarn;
- (c) cooling the filaments; and
- (d) drawing the filaments at a speed of greater than 3000 meters per minute to produce filaments having a filament denier greater than 1, and a yarn denier greater than 210.

Preferably, the filaments are drawn at a draw ratio of about 1.1 to about 4.0.

Preferably, the poly(trimethylene terephthalate) has an intrinsic viscosity of about 0.95 to about 1.10.

The drawn filaments can be bulked and/or entangled. They can be bulked to form 3-dimensional curvilinear crimp therein. Preferably, the bulking comprises blowing and deforming the filaments in a hot-fluid jet bulking unit.

According to another aspect, a process comprises:

- (a) extruding molten poly(trimethylene terephthalate) polymer having an intrinsic viscosity in the range of about 0.95 to about 1.10, a water content of less than about 100 ppm, a number average molecular weight of about 26500 to about 50000 and a melt viscosity of about 350 to about 1000 Pascals at 250°C and 48.65 per second shear rate through a spinneret to form filaments;
- (b) converging the filaments into yarn;
- (c) cooling the extruded filaments;
- (d) coating the cooled filaments with a spin finish; optionally pre-intermingling the filaments;
- (e) optionally heating the coated filaments to a temperature greater than the glass transition temperature of the polymer filaments, but less than about 200°C;
- (f) drawing the optionally heated filaments at a speed of greater than 3000 meters per minute to produce filaments having a filament denier greater than 1 and yarn having a yarn denier greater than 210;
- (g) bulking the drawn filaments such that the filaments are blown and deformed in three dimensions with a hot bulking fluid to form bulked continuous filaments having random 3-dimensional curvilinear crimp;
- (h) cooling the bulked continuous filaments to a temperature less than the glass transition temperature of the polymer filaments; and
- (i) entangling the bulked continuous filaments.

Preferably, the bulked continuous filaments are entangled before the cooling. In another aspect, the filaments can be ply-twisted and heat set into a yarn. The ply-twisted, heat-set yarn can be made into carpet.

BRIEF DESCRIPTION OF THE FIGURE

The drawings are provided for illustration purposes only, and are not intended to limit the scope of the present invention.

Figure 1 schematically illustrates a chip dryer and melt extruder system; and

Figure 2 schematically illustrates a spinning configuration useful in this invention.

DETAILED DESCRIPTION

Unless stated otherwise, all percentages, parts, ratios, etc., are by weight. Trademarks are shown in upper case.

All patents, patent applications, and publications referred to herein are incorporated by reference in their entirety.

Further, when an amount, concentration, or other value or parameter is given as either a range, preferred range or a list of upper preferable values and lower preferable values, this is to be understood as specifically disclosing all ranges formed from any pair of any upper range limit or preferred value and any lower range limit or preferred value, regardless of whether ranges are separately disclosed. Where a range of numerical values is recited herein, unless otherwise stated, the range is intended to include the endpoints thereof, and all integers and fractions within the range. It is not intended that the scope of the invention be limited to the specific values recited when defining a range.

In accordance with a first aspect of the present invention, a process comprises:

- (a) spinning molten poly(trimethylene terephthalate) polymer having a number average molecular weight of at least about 26500 and a melt viscosity of at least about 350 Pascals at 250°C and 48.65 per second shear rate;
- (b) converging the filaments into yarn;
- (c) cooling the filaments; and
- (d) drawing the filaments at a speed of greater than 3000 meters per minute to produce filaments having a filament denier greater than 1 and yarn having a yarn denier greater than 210.

The filaments can be coated with a spin finish and, optionally, preintermingled. Preferably, the process further comprises bulking the drawn

filaments. The drawn filaments can be bulked to form 3-dimensional curvilinear crimp therein. Preferably, the bulking comprises blowing and deforming the filaments in a hot-fluid jet bulking unit.

5 Preferably, the process further comprises entangling the filaments.

According to a further aspect in accordance with the present invention, a process comprises:

- 10 (a) extruding molten poly(trimethylene terephthalate) polymer having an intrinsic viscosity in the range of about 0.95 to about 1.10, a water content of less than about 100 ppm, a number average molecular weight of about 26500 to about 50000 and a melt viscosity of about 350 to about 1000 Pascals at 250°C and 48.65 per second shear rate through a spinneret to form filaments;
- 15 (b) converging the filaments into yarn;
- (c) cooling the extruded filaments;
- (d) coating the cooled filaments with a spin finish; optionally pre-intermingling the filaments;
- 20 (e) optionally heating the coated filaments to a temperature greater than the glass transition temperature of the polymer filaments, but less than about 200°C;
- (f) drawing the optionally heated filaments at a speed of greater than 3000 meters per minute to produce filaments having a filament denier greater than 1 and yarn having a yarn denier greater than 210;
- 25 (g) bulking the drawn filaments such that the filaments are blown and deformed in three dimensions with a hot bulking fluid to form bulked continuous filaments having random 3-dimensional curvilinear crimp;
- (h) cooling the bulked continuous filaments to a temperature less than the glass transition temperature of the polymer filaments; and
- 30 (i) entangling the bulked continuous filaments.

As noted, the bulked continuous filaments can be entangled before the cooling.

According to a further aspect, the filaments are ply-twisted and heat set into yarn. Carpet can be made from the ply-twisted and heat-set yarn.

With specific reference to Fig. 1 of the drawing, poly(trimethylene terephthalate) chips are loaded into dryer 10 to be dried. The intrinsic viscosity of the poly(trimethylene terephthalate) is preferably about 0.95 to about 1.10 dl/g. The intrinsic viscosity can be about 0.98 to about 1.04 or about 1.00 to about 1.02. Preferably, the number average molecular weight is at least about 26500, more preferable at least about 27500, most preferably, at least about 29000. Preferably, the number average molecular weight is up to about 50000, more preferably up to about 45000, most preferably up to about 40000. Preferably, the melt viscosity of the polymer is at least about 350, more preferably at least about 400, even more preferably at least about 450 and most preferably at least about 500 Pascals at 250°C and 48.65 per second shear rate. Also preferably, the melt viscosity is up to about 1000, more preferably up to about 900, even more preferably up to about 800 and most preferably up to about 700 Pascals at 250°C and 48.65 per second shear rate.

Drying is preferably carried out at about 80° C. or higher and about 180° C. or lower, most preferably at about 150° C. The poly(trimethylene terephthalate) chips are preferably dried until the moisture content is less than 100 ppm, more preferably about 50 ppm or less, and most preferably about 40 ppm or less. Drying time should be as long as required to reach the desired moisture content, preferably about 4 to about 10 hours, more preferably about 6 to about 8 hours. The operator should keep the moisture level steady in order to maintain consistent melt viscosity. Commercially available dehumidifiers can be used. Dry nitrogen, air or other inert gasses can be used. When the moisture content is at the desired level at the dryer exit, remelting is started.

The dried chips are fed to an optional chip metering screw 12 and are metered in to the remelter throat 14.

The metering screw is optional since the screw can be used to control the amount of chips used. A chip metering screw is normally used with a screw remelter. Any commercially available metering screw can be used.

5 By "remelter throat" reference is being made to a pipe connecting the metering screw and the remelter.

The remelter can be any suitable single or twin screw extruder. A nitrogen purge can be used to prevent oxygen from being carried along with the chips into the remelter. This will reduce oxygen-caused polymer degradation.

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Remelting is preferably carried out at about 200°C. or higher, preferably at least about 235° C., more preferably at least about 245° C., and at about 280° C. or lower, preferably about 270° C. or lower, more preferably about 265° C. or lower. At temperatures above 280° C., the undesirable byproduct acrolein is generated.

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Polymer is fed to optional transfer line pump 20, which provides sufficient pressure (about 2250-3000 psig) to overcome losses in the transfer line 22, provide constant feed rate, and provide sufficient pressure to feed the polymer to the spin pack metering pump 24. Any suitable pump may be used.

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Polymer temperature should be monitored and controlled, using techniques well within the skill of the art, to prevent polymer degradation and possible generation of irritating and/or toxic byproducts. Transfer line 22 is, preferably, surrounded by an outer pipe (not shown), which provides an outer jacket for the transfer line. The outer jacket can contain heat transfer fluid to help maintain the temperature of the polymer within acceptable limits. The temperature of the polymer transfer line 22 is preferably kept at least at about 220° C., more preferably at least at about 230° C., most preferably at least about 240° C. The temperature can be up to about 265° C., preferably up to about 260 ° C., most preferably up to about 255° C. By way of non-limiting example, the heat transfer fluid in the jacket could be paraffin kept, preferably, below 250° C.

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Polymer holdup time in transfer pipe 22 should be kept at a minimum, for example, below 20 minutes, preferably below 10 minutes, most preferably below 2 minutes. This can be accomplished, for example, by reducing the length and/or diameter of the piping and/or increasing throughput by using a booster pump.

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The metering pump 24 meters the polymer composition to the spinneret or die 26.

With reference to Figure 2, the polymer is extruded through the spinneret or die 26 to form filaments 2. Spun filaments are cooled in cooling zone 3 by a radial flow or cross flow of gas to below the polymer glass transition temperature. A spin finish or oil can be applied to the solidified filaments by finish applicator 4. Following the finish application and prior to the meter roll the filaments can be treated with turbulent air in the optional preintermingling device 5 to even out the finish on the filaments.

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The polymer is extruded through the spinneret or die at a temperature of at least about 200° C., preferably at least about 235° C., more preferably at least about 245° C., and up to about 275° C., preferably up to about 270° C., more preferably up to about 265° C.

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The spin pack metering pump and spinneret or die may be heated through conventional means (e.g., Dow fluid or hot oil).

The throughput is a function of the number of spin positions and typically is anywhere from about 2 pounds/hour (about 0.9 kg/hour) to commercial scales of about 2,000 pounds/hour (about 907 kg/hour) to about 3,000 pounds/hour (about 1,361 kg/hour) per spinning machine (i.e., per one remelter) or higher.

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The cooling zone 3 cools the filaments by a radial flow or cross flow of gas, typically humidified air at a temperature preferably of about 10° C. or above and preferably about 30° C. or below applied at about 0.2 m/sec or more and about 0.8 m/sec or less. As shown, the filaments are converged into yarn at roller 6.

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The filaments are then drawn by use of a supplying roller 6 and a set of drawing rollers 7. The filaments are preferably drawn at a draw ratio of about 1.1 to about 4.0. The draw ratio can be about 1.2 to about 3.0 or even 1.4 to 2.2.

5 The filaments can then be crimped through a bulking unit 8 with a texturing nozzle after the filaments are passed through the drawing rollers 7. The filaments can then be cooled through a cooling drum 9, and passed through intermingler 11 via roller 17, where the filaments are entangled. Thereafter, the filaments are wound with the use of a wind-up machine 15 via roller 13 and a yarn
10 guide 16.

In accordance with the present invention, the filaments are drawn at a speed of greater than 3000 meters per minute (m/min.). The draw speed can be greater than 3500 m/min., greater than 4000 m/min., greater than 5000 m/min., at
15 least 5100 m/min. or even at least 5500 m/min.

The draw ratio of the filaments is controlled by adjusting the speeds of the supply roller 6 and/or draw rolls 7 until the break elongation of the filaments is preferably at least about 10%, more preferably at least 20% and preferably no more
20 than about 90%, more preferably no more than 70%.

The drawn filament denier is greater than 1, preferably at least 3, more preferably at least 10, most preferably at least about 15 dl/g. The yarn denier is preferably greater than 210, more preferably at least about 250, even more
25 preferably at least about 500 and most preferably at least about 1000.

A jet-bulking unit 8 where the filaments can be blown and deformed in three directions with hot bulking fluid such as air or steam can be used in practicing the invention. A suitable unit is described in U.S. Pat. No. 3,525,134, the disclosure
30 of which is hereby incorporated herein by reference.

In the bulking unit described in U.S. Pat. No. 3,525,134, the filaments are both bulked and entangled. When other bulking units are used, a separate

entangling step may be necessary prior to the windup. Any method common in the trade may be used to entangle the yarn.

The resultant BCF yarn, having randomly spaced 3-dimensional curvilinear crimp, is then preferably cooled below the glass transition temperature of the filaments (approximately 45-50° C.) while the yarn is in a state of approximately 0 gpd tension so as not to pull out a significant amount of crimp. Cooling may be accomplished by a variety of commercially available means, preferably by air or water flow, spray or mist.

Using methods known in the art, the filaments can be ply-twisted and heat set into yarn. The yarn can then be made into carpet. Of course, other uses will readily occur to one of ordinary skill in the art having the benefit of the present disclosure. By way of example, the yarn of the present invention could also be used in rugs, woven tiles, automotive interiors and fabrics.

Experimental

Conditioning

Poly(trimethylene terephthalate) (3GT) resins were dried at 120 °C for 50 hours under vacuum with a heated, dry nitrogen sweep using a VWR Model 1430M vacuum oven. The moisture level in the dried resins was measured at 180 °C with 10 minute delay time using a Mitsubishi Moisture Analyzer Model CA100 with a Vaporizer Model VA100. After drying, the moisture levels in the 3GT Sample 1 and 3GT Sample 2 were 38 and 40 ppm, respectively.

Procedures

The melt stability and melt viscosity were measured at 250 and 260 ± 0.1 °C using a Dynisco LCR 7002 capillary rheometer with a 1 mm diameter, 30:1 L/D, 180 ° entrance angle die in accordance with test method ASTM D3835-02.

The melt stability was measured following procedure 10.8.1 ASTM D3835-02. A constant rate test at 48.6 s^{-1} was used with a delay time of at least 1200 seconds. Extrudate samples were collected at 40, 120, 180, 250, 360, 600, 900, and 1200 seconds. The Goodyear IV of the as-received resins and extrudates were measured in 50/50 wt% trifluoroacetic acid/dichloromethane at 19°C and a concentration of 0.4 g/dl using a Viscotek Forced Flow Viscometer Model Y-900, V5.7.

The melt viscosity was measured following procedure 10.8.2. ASTM D3835-02. A multiple rate test with software detection of steady state (procedure X2) of ASTM D3835-02. was used with a melt time of 300 seconds and a shear rate of 48.6 s^{-1} repeated at the beginning, middle and end of each test. The melt viscosity stability was determined from the slope of the best-fit line through a plot of the repeated viscosity values versus dwell time (procedure X1.4) of ASTM D3835-02. The melt viscosity stability was used to correct the data at each shear rate to zero dwell time.

Melt Stability

Goodyear IV versus time from the extrudate samples is shown in Table 1. Both 3GT resins degrade with time at the test temperatures. The initial rapid loss up to $\sim 500 \text{ s}$ is believed to be due to hydrolysis. At longer times ($> 500 \text{ s}$), the loss in IV is likely a result of thermal degradation. The rate of IV loss is about the same in both resins.

TABLE 1

| Time | 3GT | 3GT | 3GT | 3GT |
|------|--------|--------|--------|--------|
| (s) | Sample | Sample | Sample | Sample |
| | 1 | 1 | 2 | 2 |
| | 250 °C | 260 °C | 250 °C | 260 °C |
| 0 | 1.031 | 1.031 | 0.936 | 0.936 |
| 40 | 1.016 | 1.014 | 0.928 | 0.926 |
| 120 | 1.006 | 1.000 | 0.927 | 0.897 |
| 180 | 1.004 | 0.985 | 0.914 | 0.897 |
| 250 | 0.987 | 0.980 | 0.895 | 0.879 |
| 360 | 0.980 | 0.960 | 0.884 | 0.858 |
| 600 | 0.963 | 0.932 | 0.874 | 0.849 |
| 900 | 0.943 | 0.908 | 0.854 | 0.827 |
| 1200 | 0.940 | 0.897 | 0.847 | 0.814 |

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Melt Viscosity

10 Melt viscosity versus shear rate is shown in Table 2. The viscosity of the 3GT Sample 1 is higher compared to the 3GT Sample 2, consistent with a higher Goodyear IV.

Table 2.
3GT Sample 1 - Corrected Melt Viscosity

| Shear Rate (s ⁻¹) | 250 °C | | | | | 260 °C | | | | |
|----------------------------------|--------|--------|--------|---------------|-----------|--------|--------|--------|---------------|-----------|
| | Test 1 | Test 2 | Test 3 | Avg (Pa.s) | CV (%) | Test 1 | Test 2 | Test 3 | Avg (Pa.s) | CV (%) |
| 24.32 | 636.8 | 639.9 | 668.6 | 648.4 | 2.7 | | | | | |
| 48.65 | 621.8 | 623.9 | 634.5 | 626.7 | 1.1 | 495.6 | 505.0 | 499.9 | 500.2 | 0.9 |
| 72.97 | 612.4 | 612.0 | 618.2 | 614.2 | 0.6 | 492.5 | 494.5 | 493.0 | 493.4 | 0.2 |
| 97.29 | 584.1 | 603.0 | 608.8 | 598.6 | 2.2 | 484.9 | 485.0 | 487.1 | 485.7 | 0.3 |
| 121.61 | 586.9 | 585.3 | 594.5 | 588.9 | 0.8 | 476.1 | 476.4 | 479.7 | 477.4 | 0.4 |
| 182.42 | 556.9 | 541.8 | 564.9 | 554.5 | 2.1 | 457.9 | 458.5 | 459.4 | 458.6 | 0.2 |
| 243.23 | 531.6 | 535.7 | 540.9 | 536.1 | 0.9 | 441.4 | 441.3 | 437.9 | 440.2 | 0.5 |
| 364.84 | 492.3 | 494.6 | 495.7 | 494.2 | 0.4 | 412.0 | 410.1 | 411.2 | 411.1 | 0.2 |
| 486.45 | | | | | | 387.9 | 390.4 | | 389.2 | 0.5 |

3GT Sample 2 - Corrected Melt Viscosity

| Shear Rate (s ⁻¹) | 250 °C | | | | | 260 °C | | | | |
|----------------------------------|--------|--------|--------|---------------|-----------|--------|--------|--------|---------------|-----------|
| | Test 1 | Test 2 | Test 3 | Avg (Pa.s) | CV (%) | Test 1 | Test 2 | Test 3 | Avg (Pa.s) | CV (%) |
| 24.32 | 314.8 | 317.3 | 308.5 | 313.6 | 1.4 | 258.4 | 247.3 | 273.8 | 259.8 | 5.1 |
| 48.65 | 308.9 | 309.1 | 317.4 | 311.8 | 1.6 | 251.8 | 243.7 | 262.1 | 252.5 | 3.0 |
| 72.97 | | 298.1 | 300.6 | 299.4 | 0.6 | 241.2 | 241.5 | 260.2 | 247.6 | 3.6 |
| 97.29 | 300.2 | 300.1 | 303.8 | 301.4 | 0.7 | 247.2 | 238.4 | 258.8 | 248.1 | 3.4 |
| 121.61 | 297.1 | 294.6 | 306.9 | 299.5 | 2.2 | 247.1 | 234.6 | 255.9 | 245.9 | 3.5 |
| 182.42 | | | | | | 242.9 | 230.9 | 250.6 | 241.5 | 3.3 |

Size Exclusion Chromatography Method to Measure Molecular Weight

Distribution in Polymers Soluble in HFIP

Polymer samples were under dissolution for 2 hours in mobile phase solvent at 50° C with moderate agitation (Automatic sample preparation system PL 260™ from Polymer Laboratories). All concentrations are in milligram per milliliter (mg/ml). Mobile phase solvent was 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) with 0.01 molar sodium trifluoroacetate.

Polymer solutions were injected into size exclusion chromatography system. The system included size exclusion chromatography system Model Alliance

2690TM from Waters Corporation (Milford, MA), with a Waters 410TM refractive index detector (Differential Refractive Index) and Viscotek Corporation (Houston, TX) Model T-60ATM dual detector module incorporating static right angle light scattering and differential capillary viscometer detectors. Columns for separation were Two
 5 Shodex GPC HFIP-80MTM styrene-divinyl benzene columns with exclusion limit 2 x 10⁶ and 8,000/30cm theoretical plates. Chromatographic conditions were at 35°C temperature, 1.00 ml/min flow rate, 0.1 ml injection volume and 50 minute run time.

Software used for data reduction was Trisec® Triple Detector SEC³
 10 version 3.0 by Viscotek. Data reduction method was via triple detection method incorporating data from all three detectors: refractometer, viscometer and light scattering photometer (right angle). Flory-Fox equation is used for angular asymmetry light scattering correction. No column calibration was involved in data processing. Sample concentration for 3GT polymers in HFIP was verified independently based
 15 on refractive index increment (dn/dc) = 0.235. Number average molecular weight was calculated and reported as shown in Table 3.

Table 3.
Number Average Molecular Weight

| Resin | Measurement 1 | Measurement 2 | Average |
|--------------|---------------|---------------|---------|
| 3GT Sample 1 | 33200 | 33200 | 33200 |
| 3GT Sample 2 | 26800 | 25300 | 26050 |

Example 1 (3742 mpm Draw Roll Speed)

25 Poly (trimethylene terephthalate) polymer (3GT, PTT) in chip form, specifically 3GT Sample 1, was dried in a rotary dryer. Drying was done under vacuum at 160 degrees centigrade (° C) for 6 hours, cooled with nitrogen gas to 25° C and stored in a sealed vessel to maintain a moisture level less than 50 ppm. For remelting, the chip was fed to a dry nitrogen supply hopper at room temperature and
 30 then gravity fed into the throat of the extruder. An alternative method is to have a drier mounted above the extruder and continuously dry chip at 160° C for 6-8 hours using dry nitrogen or air. A dry nitrogen purge was located at the extruder throat to remove oxygen from the down coming chip when using dry air.

The single screw extruder was set at:

| | | |
|----|----------------|--------|
| 5 | Zone1 | 230° C |
| | Zone 2 | 240° C |
| | Zone 3 | 250° C |
| | Zone 4 | 250° C |
| | Zone 5 | 250° C |
| 10 | Extruder speed | 14 rpm |
| | Melt Pressure | 80 bar |

The extruder discharge melt temperature was 250° C The transfer line and spin beam temperature was maintained at about 250° C. The melted polymer was fed to a 2-pack spin beam. In the spin beam metering gear pumps provided 76 bar pressure to the spin pack. Each pump had a capacity of 30 cubic centimeters per revolution (cm^3/rev). The pumps were run at 12.10 rpm. Each pack had a 1 layer metal screen filter with a screen mesh size of 10,000 M/ cm^2 . The spinnerets each had 68 trilobal (Y) holes with capillary diameter of 0.35x0.66 mm with a length of 0.6 mm.

The extruded or spun filaments were quenched with 18° C air maintained at 80% humidity with a quench zone length of 1600 mm. Average air cross flow was 0.35 meters/second (m/s). The filaments were pulled down through a one floor high interfloor tube (part of a 3 floor machine) to a Neumag Bulk Continuous Filament (BCF) spinning machine. At the bottom of the interfloor tube two sets of 68 filaments were converged using finish applicators. The contact width of the upper applicators was 5 millimeters (mm) and the lower reversed finish applicators were 2 mm. Two 4 stream 0.8 cm^3/rev finish pumps set at 35 rpm pumped 18 % standard finish to the finish applicators.

The threadlines were led onto an inlet godet (roller) with a surface speed of 1950 meters per minute (m/min.), then, onto a metering godet duo set at 40° C with a surface speed of 1970 m/min.. The filaments were drawn in space by advancing to a set of enclosed heated duos set at 165° C with a surface speed of 3742 m/min. The filaments were heated by the godets fed into a Neumag texturing chamber that had a lamella cone of 3 / 4.5 mm and length of 80 mm. 18 lamella pieces formed the cone. Hot air set at 7.0 bar and 225° C impinged on the yarn

bundles. The lamella exhaust cone had a vacuum setting of –70 millibars (mbar). The textured or bulked yarn flowed out of the bottom of the chamber and piddled onto a cooling drum with a surface speed of 60 m/min.

5 The cooled threadlines were removed from the cooling drum with a godet with a surface speed of 3010 m/min. From the godet the threadlines went through a tacking or intermingling box that had an air jet with a yoke width and diameter of 6 mm. The threadlines were impinged with an air pressure of 5.5 bar. The correct tension was controlled by an exit godet with a surface speed of 3030
10 m/min. This godet isolated the winding tension from the required tacking tension.

The threadlines were led to a two-cot winder that takes a tube diameter of 79 millimeters (mm). The drive roll or pressure roll (set at 100 newtons (N)) surface speed was 3015 m/min., which produced a winding tension of around 150
15 grams. The traversing stroke was 250 mm and was run at speed to produce a 13-degree winding angle. The traversing mechanism was modulated with an amplitude of 0.1% at 0.1 / second. The final package diameter was 215 mm producing a package weight of 5.1 kilograms.

20 Textile measurements were:

| | | |
|----|------------------------|------|
| | Denier | 1242 |
| | Tenacity, gm/den | 2.63 |
| | Elongation, % | 50 |
| | Modulus, gm/den | 13.3 |
| 25 | TYT, % TR ¹ | 16. |
| | TYT, % CO ² | 14.5 |
| | TYT, % FS ³ | 2.4 |

¹ TYT=Bulk measurement instrument of Lawson-Hemphill Electron

30 Yarn Tester Model TYT-EW, %TR=Total Retraction

² %CO=Crimp Out

³ %FS=Fiber Shrinkage

Example 2 (4100 m/min. Draw Roll Speed)

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Poly (trimethylene terephthalate) polymer (3GT, PTT) in chip form, specifically 3GT Sample 1, was dried in a rotary dryer. Drying was done under

vacuum at 160 degrees centigrade (° C) for 6 hours, cooled with nitrogen gas to 25° C and stored in a sealed vessel to maintain a moisture level less than 50 ppm. For remelting, the chip was fed to a dry nitrogen supply hopper at room temperature and then gravity fed into the throat of the extruder. An alternative method is to have a drier mounted above the extruder and continuously dry chip at 160° C for 6-8 hours using dry nitrogen or air. A dry nitrogen purge was located at the extruder throat to remove oxygen from the down coming chip when using dry air in the drier.

The single screw extruder was set at:

| | | |
|----|----------------|--------|
| 10 | Zone1 | 230° C |
| | Zone 2 | 240° C |
| | Zone 3 | 250° C |
| | Zone 4 | 250° C |
| | Zone 5 | 250° C |
| 15 | Extruder speed | 15 rpm |
| | Melt Pressure | 80 bar |

The extruder discharge melt temperature was 250° C. The transfer line and spin beam temperature was maintained at 250° C. The melted polymer was fed to a 2-pack spin beam. In the spin beam metering gear pumps provided 79 bar pressure to the spin pack. Each pump had a capacity of 30 cm³/rev. The pumps were run at 13.26 rpm. Each pack has a 1 layer metal screen filter with a screen mesh size of 10,000 M/cm². The spinnerets each have 68 trilobal (Y) holes with capillary diameter of 0.35x0.66 millimeters (mm) with a length of 0.6 mm.

The extruded or spun filaments were quenched with 18° C air maintained at 80% humidity with a quench zone length of 1600 mm. Average air cross flow was 0.25 meter per second (m/s). The filaments were pulled down through a one floor high interfloor tube (part of a 3 floor machine) to a Neumag spinning machine. At the bottom of the interfloor tube the two sets of 68 filaments were converged using finish applicators. The contact width of the upper applicators was 5 mm and the lower reversed finish applicators were 2 mm. Two 4 stream 0.8 cm³/rev finish pumps set at 40 rpm pumped P-7050T 18 % Fiber Solutions finish to the finish applicators.

The threadlines were led onto an inlet godet with a surface speed of 2390 m/min.. Then, onto a metering godet duo set at 40° C with a surface speed of

2400 m/min.. The filaments were drawn in space with no assist by advancing to a set of enclosed heated duos set at 165° C with a surface speed of 4100 m/min. The filaments were heated by the godets fed into a Neumag texturing chamber that had a lamella cone of 3 / 4.5 mm and length of 80 mm. 18 lamella pieces formed the cone.

- 5 Hot air set at 7.5 bar and 225° C impinged on the yarn bundles. The lamella exhaust cone had a vacuum setting of -95 m/bar. The textured or bulked yarn flowed out of the bottom of the chamber and piddled onto a cooling drum with a surface speed of 65 m/min.

- 10 The cooled threadlines were removed from the cooling drum with a godet with a surface speed of 3300 m/min.. From the godet the threadlines went through a tacking or intermingling box that had an air jet with a yoke width and diameter of 6 mm. The threadlines were impinged with an air pressure of 7.0 bar. The correct tension was control by an exit godet with a surface speed of 3340
15 m/min.. This godet isolated the winding tension from the required tacking tension.

- The threadlines were led to a two-cot winder that took a tube diameter of 79 mm. The drive roll or pressure roll (set at 100 N) surface speed was 3305 m/min., which produces a winding tension of around 150 grams. The traversing
20 stroke was 250 mm and was run at speed to produce a 13-degree winding angle. The traversing mechanism was modulated with an amplitude of 0.1% at 0.1 / second. The final package diameter was 215 mm producing a package weight of 5.1 kilograms.

- 25 Textile measurements were:
- | | |
|------------------|------|
| Denier | 1212 |
| Tenacity, gm/den | 2.71 |
| Elongation, % | 51 |
| Modulus, gm/den | 13.1 |
| TYT, % TR | 16.4 |
| TYT, % CO | 13.8 |
| TYT, % FS | 3.0 |

¹ TYT=Bulk measurement instrument of Lawson-Hemphill Electron

- 35 Yarn Tester Model TYT-EW, %TR=Total Retraction

² %CO=Crimp Out

³ %FS=Fiber Shrinkage